



Standard Test Method for Measurement of Transition Temperatures of Petroleum Waxes by Differential Scanning Calorimetry (DSC)¹

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^{ε1} NOTE—Section 15 was added editorially in June 1995.

1. Scope

1.1 This test method measures the transition temperatures of petroleum waxes, including microcrystalline waxes, by differential scanning calorimetry (DSC). These transitions may occur as a solid-solid transition or as a solid-liquid transition.

1.2 The normal operating temperature range extends from 15°C to 150°C (Note 1).

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.4 The values stated in SI units are to be regarded as the standard.

2. Referenced Documents

2.1 ASTM Standards:

D 87 Test Method for Melting Point of Petroleum Wax (Cooling Curve)²

D 1160 Test Method for Distillation of Petroleum Products at Reduced Pressures²

D 3418 Test Method for Transition Temperatures of Polymers by Thermal Analysis³

E 472 Practice for Reporting Thermoanalytical Data⁴

E 473 Terminology Relating to Thermal Analysis⁴

E 474 Test Method for Evaluation of Temperature Scale for Differential Thermal Analysis⁵

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *Differential Scanning Calorimetry (DSC)*—A technique in which the difference in energy inputs into a substance and a reference material is measured as a function of tempera-

ture, while the substance and a reference material are subjected to a controlled temperature program. The record is the DSC curve. Two modes, power-compensation DSC and heat-flux DSC, can be distinguished depending on the method of measurement used. For additional background information refer to Practice E 472, Terminology E 473, and Test Method E 474.

4. Summary of Test Method

4.1 Separate samples of petroleum wax and a reference material or blank (empty sample container) are heated at a controlled rate in an inert atmosphere. A sensor continuously monitors the difference in heat flow to the two samples. The DSC curve is a record of this difference versus temperature. A transition in the wax involves the absorption of energy relative to the reference, resulting in an endothermic peak in the DSC curve. While the transition occurs over the temperature range spanned by the base of the peak, the temperature associated with the peak apex is designated the nominal transition temperature (Note 1).

NOTE 1—Test Method D 87 also monitors energy transfer between wax and a standard environment. The highest temperature DSC transition may differ from the melting point because the two methods approach the solid/liquid phase transition from different directions.

5. Significance and Use

5.1 DSC is a convenient and rapid method for determining the temperature limits within which a wax undergoes during transitions. The highest temperature transition is a solid-liquid transition associated with complete melting; it can guide the choice of wax storage and application temperatures. The solid-solid temperature transition is related to the properties of the solid, that is, hardness and blocking temperature.

NOTE 2—For a relatively narrow cut petroleum wax, the lowest transition will be a solid-solid transition. A narrow cut wax is one obtained by deoiling a single petroleum distillate with a maximum range of 120°F between its 5 % and 95 % vol in accordance with Test Method D 1160 boiling points (converted to 760 torr). The DSC method cannot differentiate between solid-liquid and solid-solid transitions. Such information must be predetermined by other techniques. In the case of blends, the lower temperature transition may be envelopes of both solid-liquid and solid-solid transitions.

5.2 Since petroleum wax is a mixture of hydrocarbons with

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² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 08.02.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Discontinued; see 1985 Annual Book of ASTM Standards, Vol 14.02.